DATA QUALITY REPORT SERIES

QUALITY CONTROL PROCEDURES AND OBJECTIVES

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Ministry of the Environment

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LABORATORY SERVICES BRANCH

DATA QUALITY REPORT SERIES

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Laboratory Services Branch

Quality Control Program

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QUALITY CONTROL

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INTRODUCTION

The Data Quality Report Series was initiated to facilitate the documentation and formalization of quality control procedures within the Laboratory Services Branch of the Ontario Ministry of the Environment.

In this report on Quality Control Procedures and Objectives, an effort is made to define the objectives of a data quality assurance program, and to outline in broad terms the specific control areas which must be considered in implementing and formalizing such a program.

Also included is a summarization of existing control mechanisms as reported by analysts in the various sections of the Laboratory Services Branch. Quality Assurance is always a dynamic process. Any attempt to document procedures invariably results in review and revision. Hopefully this report will provide further impetus in this direction.

I wish to acknowledge the encouragement and help I have received from the managers, chiefs, and supervisors in the Laboratory Services Branch. The establishment of the position of quality assurance officer has provided the time, and freedom from routine workload pressures required to permit a critical review of existing quality control procedures.

QUALITY CONTROL OBJECTIVES

Quality control is essentially a state of mind which encourages one to investigate existing procedures. Are they efficient? Do they do the job required? There is a fine balance required between productivity and quality of product. Neither has priority.

Many will comment that quality control is expensive and cuts productivity and yet there is nothing further from the truth. Proper quality control simplifies decision making, prevents waste effort and minimizes the possibility of error. Therefore, if properly applied, it should improve output.

There are those who claim that quality control requires analysis of a specific number of duplicates per day or specifies the number of standards or control samples to be run. In fact the better analysts find it difficult to describe their control activities because everything they do relates in some way to data quality assurance.

However, human nature being what it is, there is a need to document these activities in order to ensure they are continued, are efficient and effective, and are understood and appreciated as necessary. It is perhaps more important to document how and why the procedure was developed than to describe the procedure itself.

If a quality control program is to be effective and yet efficient, it must account for the various causes of imprecision, and relate these to the data need. The table on the following page outlines some of the sample matrices, analytical parameters and sample fractions encountered by analysts in the Laboratory Services Branch. It also lists the variety of analytical and sample preparation techniques which may be involved in order to meet the data need. Obviously there can be no single approach to quality control.

SAMPLE TYPES

Unpolluted rivers, lakes
Drinking waters
Polluted receiving waters
Municipal waste treatment
Industrial waste treatment
Digester supernatants
Digester sludges
Industrial waste organics
Leachates
Solid wastes
Sediments
Soils
Vegetation
Fish tissue
Human tissue
Hi-vol air filters

PARAMETERS

Water quality
Minerals
Nutrients
Metals
Organics
Pesticides
Mercury
Asbestos
Physical
Microbial
etc.

FRACTIONS

Particulate
Dissolved
Extractable
Free
Combined
Reactive
etc.

ANALYTICAL TECHNIQUES

Standard chemical
Microbiological
Auto Analyzer
Atomic Absorption
Emission spectroscopy
Inductively coupled plasma
Anodic Stripping voltammetry
IR spectroscopy
Gas liquid chromatography
Mass spectrometry
X-ray fluorescence
Electron-microscopy
etc.

SAMPLE PREP.TECHNIQUES

Drying, homogenization
Separation, fractionation
Filtration, extraction
Digestion, etc.

DATA NEEDS

Baseline leves or trend analysis.
Court cases.
Identify problems.
Develop contours to describe zones of influence.
Develop theories to explain decay mechanisms.
etc.

In order to demonstrate the variety of approaches taken by different analysts, statements describing the philosophy and resulting control techniques developed in the various sections of the Laboratory Services Branch have been prepared, and are included later in this report.

These procedures are still subject to review and do not necessarily reflect the final position that will be taken. The statements have been prepared in order to provide a basis for further discussion.

In outlining the general scope of an interlaboratory data quality assurance program the following objectives seem to be primary.

- The first objective must be to ensure that laboratory precision is not limiting data quality. One should avoid, if at all possible, the use of insensitive analytical systems if good low level information is required.

- The second objective is to ensure that imprecision resulting from sample inhomogeneity is not used to excuse imprecision or inaccuracy in the calibration process. Sample imprecision can be improved by multiple analysis; bias in calibration which changes from day to day cannot be removed except at the time it occurs.

- The third objective is to determine where control can be most effectively exerted to improve or maintain precision levels, and minimize the variety of controls required.

- The fourth objective, which can only be properly addressed when precision is optimized, is to determine the amount, nature and sources for bias, and thence to implement control over it.

- The fifth objective is to document and thereby formalize the control procedures required, including the data evaluation technique.

Data evaluation is a particularly weak area. Quality control has adopted many of the common statistical techniques but the tendency is towards misuse since these tools assume no prior experience or other source of information than the data at hand. At best they are too conservative in predicting outliers or unexpected behaviour. The analyst, on the other hand, knows what to expect and is looking for deviation from the law as evidence of system breakdown. In many cases statistical conclusions can be modified by this knowledge.

INTRALABORATORY CONTROL

A good data quality system requires continuing, active control procedures to permit timely response to any indication of unsatisfactory performance. The details of the analyst's response depend on the needs of the procedure being monitored, but no consistent operator response is possible if the control is applied irregularly, is not sufficient or suitable to the system, or if the control results are ignored. It is usually essential that the control package be specifically designed to achieve and maintain the desired level of performance. It is necessary that controls be applied regularly, and that some measure of their effectiveness be forthcoming, without increasing significantly the routine workload of the analyst.

There are only two major areas for control, namely precision (or freedom from variability) and accuracy (or freedom from error). Of these the most important is accuracy since, on average, variability is self-cancelling whereas bias is not.

Reproducibility in reporting results is generally limited by one of the following factors in order of increasing significance

a) electronic (instrument) noise observed under extreme range expansion.

b) inability to observe small changes in reading because of insufficient range expansion.

c) analytical noise, eg. non reproducible colour development or noisy reagent background, independent of sample preparation procedures.

d) sample preparation noise, eg. poor reproducibility after sample digestion.

e) sample non-homogeneity.

f) poor sampling or sample handling and preservation.

Field and/or time variability do not relate in any way to precision of analysis. If a representative sample is difficult to obtain then one should recognize that a single sample or even the average of several samples may not adequately describe the object of study.

The methods available for day-to-day bias control are not always correctly applied or appreciated. The two sources of bias almost always relate to poor calibration control. One of those is in the blank or zero intercept, the other is in the recovery or slope factor relating instrumental response and concentration. (In spite of feelings to the contrary it is the response that is known, rather than the concentration, particularly after the standards have been processed through extensive preparation procedures. If the expected response is not obtained either the machine is not operating properly or the standards are wrong.)

Every practising analyst exerts some form of quality control as a routine part of his analytical duties. Whether his control procedures are necessary, sufficient, or effective, in ensuring data quality depends upon a clear statement of what constitutes good analytical practice modified by the projected data use. *

The following pages identify clearly areas which should be considered in documenting or formalizing a quality control program. In order to adequately assess the need for a particular type of control the following information should be available.

1) A statement defining the level of data quality required or achievable, relating this to the type of sample, analytical methods available, complexity of sample preparation, and the

routine function of the laboratory performing the analysis.

2) A review of the principle involved in a particular control procedure. Its advantages and disadvantages relative to other possible techniques should be discussed.

3) A general description of the control procedures used, their intent, and action to be taken.

4) A statement of the specific control procedures for specific analytical methods and instruments. Frequency of application and format for reporting the data should be defined.

The immediate goal of the Quality Assurance Office is to document, in detail, flow diagrams showing sources and types of samples, sample preparation techniques and instrumentation employed, throughout all the section of the Laboratory Services Branch, in order to facilitate definition of quality control needs. This will lead immediately to a comparison of needs versus existing practice. In cooperation with the analysts, specific quality control procedures will then be defined, and formalized.

Precision:

- ability to reproduce analytical results (not only within the same analytical batch but also from day to day).
- demonstrated by continuing routine duplicate analysis of actual samples, (approx. 5 to 10% of workload)
  a) split at lab, analyzed same run
  b) split at lab, analyzed different run
- may be extended to include field sampling and transportation effects by means of separate field samples submitted blind to the lab, however there must be evidence that the samples are in fact replicates. (Failure to obtain reproducible results may reflect real sample differences.)
- because precision depends on sample homogeneity, failure to reproduce results must be related to the sample type. When analyzing a wide variety of samples, an 'out-of-control' duplicate may not be evidence of an out-of-control analytical system.
- imprecision must be defined relative to data needs but should not be tolerated if more precise data can be readily achieved without sacrificing analytical throughput time or efficiency.
- passive monitoring of precision is usually sufficient when using routine procedures for which performance has been adequately documented.
- when data is being averaged, imprecision may be acceptable provided that the technique is sufficiently sensitive and unbiased.

Calibration:

- ability to relate a change in instrumental response to a corresponding change in mass or concentration.
- is achieved by means of prepared standardized solutions covering the range of instrument response
  a) at two points (if system known linear and stable)
  b) at several points (if system imprecise or curved)
- is confirmed by means of paired control standards or samples chosen to monitor for the specific type of systematic bias expected between runs (with or without sample preparation included)
  a) two high level controls (slope bias only)
  b) two low level controls (blank bias only)
  c) one high, one low, level control
- the primary criteria is stability of response to a 'known' concentration (Instrumental response must be recorded). Calibration standards should be permitted the same variability as expected for samples. They should not be made to read exactly by adjusting range expansion (or Technican standard/cal) controls. Adjustment is made only if the control samples are 'out-of-control'.
- precision is required in order to detect and minimize between-run changes, therefore standards should be analyzed directly with minimal or no sample preparation, eg. undigested.
- is completed before the analytical run commences. Emphasis is placed on controlling bias and confirming linearity and in-run stability.
- active control is required if there is a significant change in
  a) instrumental response
  b) curvature, or
  c) evidence of bias.

Background:

- the need to establish an absolute zero reference by use of appropriate blank materials.
- clarification of procedures, and identification of sources of blank error is required. The role of field (container) blanks needs attention.

NOTE: whereas slope calibration is not expected to change, when significant blanks are being observed there is no reason to believe that the blank will not change. However, any action to be taken on the basis of a change in blank must be well supported to avoid over correction.

Recovery:

- ability to detect and correct for errors introduced during sample preparation or resulting from interferences or matrix (background) effects.
- checks are required whenever the sample matrix is different from that of the calibration standards, interference is suspected, or complex sample preparation procedures are employed.
- check is performed by applying the total analytical process to
  a) simple (pure, easily recoverable, soluble) forms of the (molecular, etomic) parameter in standard solution.
  b) complex (pure, recoverable after sample treatment) forms (eg. organo-metallics, organic complexes) in standard solutions.
  c) mineralized or biologically incorporated forms including the more refractory or less easily recoverable species, in the form of reference materials.
- since failure to recover may be the result of
  a) loss during extraction or leaching by some escape mechanism (volatility; precipitation).
  b) inefficiency of the sample preparation procedure (inability to break down the matrix to release the parameter).
  c) insensitivity of the analytical process to some forms of the parameter.
  d) background ionic strength, pH, solution density, or other matrix effects.
  e) interferences which react in a similar way or prevent reaction of the desired constituent.

  each recovery technique must be modified to meet the specific type of problem encountered.
- routine confirmation of recovery is monitored by paired control samples (eg. in-house reference fish, vegetation or soil samples for which a history of analysis is available.
- other checks would include a series of digested standards, spiking of routine samples (eg. 1 in 20) or in special cases, application of the method of standard additions.

NOTE: Imprecision resulting from the various steps in the analytical chain can easily result in misinterpretation of recovery. Care must be taken to ensure that
  a) a recovery factor is not based on only one or two analyses.
  b) analytical precision is not used to excuse possible recovery failure.

- passive control based on observation of recovery trends is basic to any decision on recovery. A sudden change in recovery factor should cause the analytical process to stop. New factors should not be calculated without strong confirmatory evidence.

Data Compatibility:

- the ability to relate your results to those being reported by other analysts. The analytical techniques need not be the same since the first criteria is to obtain and demonstrate a definable (precise) pattern of deviation.
- failure to achieve this, after control over precision, calibration and recovery has been exerted, may indicate lack of specificity in one or more of the procedures being compared. Nomenclature should be changed to reflect this.
- intercomparison studies should evaluate precision, as well as relative recovery and background differences (slope and blank deviation between laboratories).
- whenever possible, natural samples, submitted on a regular continuing basis to two or more analysts, should be used to assess continuing data compatibility. It should not be assumed that data compatibility on natural samples is unachievable. Failure to obtain this objective may reflect lack of control over one or more of the areas described above.
- special studies can be initiated for specific parameters when problems are detected, in order to determine the source of difficulty.
- many intercomparisons do not include enough samples to permit evaluation for systematic error by regression.
- many intercomparisons are not successful because of lack of proper, complete, data evaluation techniques. The treatment of outliers, data with insufficient significant figures, and laboratories whose precision is not comparable to the "better" laboratories, is not consistent. Statistical treatments tend to lump all data together without considering the relative capabilities of the participants. Clarification of procedures is needed.

Specificity:

- ability to separate the desired constituent from other species which will respond similarly under the test conditions.
- some procedures are non specific by nature. Data compatibility will depend upon adequate definition and control of the test procedures. This must be reflected in the choice of nomenclature.
- may be checked by
  a) alternative analytical procedures
  b) documentation of the nature of sample types causing recovery problems
  c) testing of suspected interferants
  d) documentation of physical parameters such as retention time, line separation, particle size, colour, etc.

Accuracy:

- the ability to obtain the correct results at all times. This requires evaluation against accepted standard reference materials (SRM's). Accuracy cannot be properly demonstrated until routine documentation of continuing precision, recovery, and data compatibility is available. In-house reference materials used as a control over each analytical run are essential. When the homogeneity and stability of the control samples has been demonstrated, it is then proper to analyze both the SRM and the in-house control simultaneously in several replicates in order to establish the concentration of the in-house materials. These then can be used to control not only between-run precision but also absolute accuracy on a continuing basis.

DATA QUALITY DOCUMENTATION

As a result of requests from various Reference Groups and Subcommittees of the Great Lakes Water Quality Board of the International Joint Commission (IJC), that reviews be prepared to describe past analytical performance, some considerable effort was spent as a member of several data quality workgroups in developing and evaluating the various questionnaires required.

In attempting to design and then respond to such questionnaires it became obvious that a consistent approach must be developed for the provision and storage of analytical method reports, evaluations, critiques, internal performance checks and intercomparison data of all types. In general, laboratory files are full of raw data and reports which address many of the concerns of Data Quality but frequently this information and data is inadequately tabulated and evaluated and is not always filed in a systematic fashion for ready retrieval. Much of what is known about previous, as well as existing, techniques is not written down but rather is part of the expertise of the professional staff most closely associated with the development and application of these techniques. There is a real danger that this experience is lost whenever staff are transferred.

The following table identifies four areas which would be expected to generate reports affecting data quality assessment. The ideal quality assurance program would ensure the systematic preservation of knowledge in these areas. This is perhaps more important than documenting control procedures because while the latter become part of the unwritten laws under which the analyst functions, knowledge which justifies the practice tends to be lost.

DATA QUALITY ASSURANCE PROGRAM

OBJECTIVE: To build and maintain a central file of reports covering the following topics for each parameter and method.

A. METHODOLOGY

1. Method Development
   a) Preliminary Report
   b) Interim Report
   c) Final Report
   d) Supplementary Report
2. Annual Critiques
   a) Existing Method
   b) Alternative procedures
   c) Alternative instrumentation
3. Technical Notes
4. Parameter & Method Codes
5. Notification of Change
6. Methods Manual Updates
7. Inter-method Studies

B. IN-LAB PERFORMANCE ←

1. Method Stability Data Tables
   a) Instrument Response
   b) Blank Control
   c) Calibration Control
   d) Reagent & distilled water checks
2. Working Control Charts
3. Summary Table of Control Data
4. Quarterly Review Report
5. Tabulation of Internal Blind Checks & Results
6. Specification of Exact Control Requirements

C. EXTERNAL CHECKS

1. Youden-type two-sample controls (EPA ampoules) based on
   a) reference materials
   b) stabilized unknown natural materials
   c) 'fresh' natural materials
2. Special Study Comparison between two labs (multi-sample)
3. Duplicate field samples (on-going)
4. Long Term regular interlab comparison as part of the field program (split sample between regional and central laboratory)
5. Multi-sample multi-parameter interlab comparison for identifying slope and blank bias.

D. RELATED STUDIES

- Field effects
- container effects
- preservatives
- sample matrix
- non-lab blanks
- etc.

Methodology

Reports describing the procedure used, developmental studies carried out and problem areas encountered should be continually updated. This ensures that the technique is under regular review with respect to updated literature and instrumentation, and that relevant staff are notified of changes which may affect data quality.

In-lab Performance

Documentation of the proper operation and ongoing stability of an analytical procedure requires regular provision, charting and summarization of control data. A regular narrative report on overall performance and a discussion of problems encountered is useful.

External Checks

The proof of adequate performance comes through comparison of data with other analysts. A file of all such raw data is required including the evaluation provided by the check program originator, as well as our own evaluation. There is also a need to review these assessments regularly to summarize the various findings.

Related Studies

There is a wide variety of activities which affect overall data quality. These studies should be keyed into the parameters affected and should result in appropriate modification of technique, container use, field activities, etc. Again a common file is required for such information, including data and proof of implementation.

The Performance Report

The performance report, perhaps the single most important form of documentation, can be considered to consist of four separate components which contribute to overall performance evaluation.

a) Sample-related factors which assist in defining the performance characteristics (including such things as sensitivity) required of the methodology and the extent to which sample effects may degrade the actual performance of a routine methodology. This includes discussion of sample matrix, preservation, handling, and preparation.

b) Analytical techniques which helps to define the degree of analytical difficulty independent of sample source.

c) Instrumentation factors which document, for example, the extent to which instrumental resolution (or lack of it) is improved by means of scale expansion to meet the sensitivity requirements of the sampling program. They assist also in cross-comparison of different techniques.

d) Performance data documenting such things as calibration stability, within and between-run repeatability under controlled conditions, and precision data based on actual sample analysis.

These components are further delineated in other sections of the Data Quality Report Series as needed. However, an example of a typical performance report is shown on the following page.

PERFORMANCE REPORT

NITROGEN
Total Kjeldahl Nitrogen
mg/l N
Automated Colorimetric
CODE 20

DATA APPLIES TO LAB

EL	DW	WT	TB	LO
X			X	X

SAMPLE TYPE: River and lake.

SAMPLE PREPARATION: 4 ml of 20% H_2SO_4 are added to 50 ml of sample which is digested to white fumes, oxidized with persulphate for 20 minutes of white fumes and neutralized with NaOH to the red side of methyl red.

ANALYTICAL PROCEDURE: (initiated 6 / 67) STANDARD ALIQUOT .32 mls /min (20%)

Hypochlorite and phenate are mixed with digested sample in an automated system. An acetone catalyzed reaction at 37.5°C produces a blue indophenolic compound which is analyzed colorimetrically in a continuous flow system.

INSTRUMENTATION: Technicon AAII system, standard calibration 1.48, 630 nm, 50 mm flow cell, manual readings.

operating scale:	0 - .80 Abs
calibrated:	0 - 2.0 mg/l N
results read to nearest:	0.02
results reported to nearest:	3 sig. fig.

CALIBRATION STABILITY: The ratio s/sw = 1.2 indicates between run effects are well controlled. The instrument is stable with respect to calibration, however, the digestion recovery may affect calibration and therefore requires daily monitoring.

BLANKS: Digestion blanks due to the persulphate oxidation step vary between 0.05 - 0.08. Sample correction is based on three blanks and duplicates of a low digested standard, analyzed daily.

STANDARD DEVIATIONS: (Concentration of Controls A: 1.55 B: 0.76)

PERIOD 1975	WITHIN-RUN DUPLICATES			CONTROL DATA	
	S_{ld}	S_{md}	S_{hd}	Sw	S
Jan. - Feb.	.030		.053	.082	.083
March - Apr.	.018		.030	.038	.064
May - June	.036		.039	.030	.039
July - Aug.	.029	.035	.047	.031	.037
Sept. - Oct.	.021		.027	.022	.024
Nov. - Dec.	.017		.048	.018	.019
AVERAGE SD:	.026	.035	.037	.037	.044
RELATIVE STANDARD DEVIATION:	2.5%				
PRECISION:	.072	.097	.102	.102	.122
CONC'N LEVEL:	.20	.60	1.5		

DETECTION CRITERIA: 0.043 DETECTION LIMIT: 0.086

REMARKS:

Analyzed simultaneously with total phosphorus.

- Variation in the persulphate used in the digestion from batch to batch may cause blank problems, but atmospheric contaminations can also cause variability in the blank value.

MICROBIOLOGY QUALITY ASSURANCE PROGRAMS

Setting up quality assurance programs in a media preparation laboratory presents some unique problems. Little information is available on the problems and pitfalls involved in the preparation and storage of media, and as a result few laboratories implement a strict quality control program. In contrast, such programs are in routine application in chemistry and haematology laboratories where the determinations are quantitative and can usually be analyzed statistically. Facilitating this is the fact that reference standards may be easily prepared or obtained commercially.

Major sources of error in a microbiology laboratory include improper preparation or storage of media, equipment malfunction, inadequate cleansing or sterilization of glassware and impure water supplies. A quality assurance program should strive to ensure reliability and reproducibility of results with minimal effort and expense by monitoring these problem areas.

To implement such a program entails first of all the preparation of a list of procedures, materials and equipment which are to be subject to periodic monitoring. Useful references on this subject are papers by Russell *et al.* (1969), Glasser *et al.* (1971) and Bartlett (1975b). A routine timetable can then be set up in accordance to the priorities specific to the laboratory involved. These priorities should reflect the likelihood of deficiencies based on previous observations and the relative importance of the variable considered. All observations should be recorded in books or journals as suggested earlier in this respect.

Bartlett (1975b) in his detailed outline of a functional quality control program has indicated that high priority areas in media surveillance should include sterility checks, pH, storage restrictions and testing with stock cultures. Of less priority are depth of plates and reagent inventory. Under equipment maintenance, the items of highest priority include refrigerators, incubators, water baths, laminar air flow cabinets, autoclaves and hot air ovens. Bartlett also suggests that surveillance of microscopes, balances and glassware be given less emphasis, but stresses the high priority of maintaining a frequently updated methods manual.

Implementation of the programs must also be monitored. This is most efficiently accomplished by the preparation of a monthly surveillance report as suggested by Bartlett (1975b). This report should indicate whether or not surveillance has been conducted as scheduled, whether any deficiencies were observed and the necessary corrective action. Detail can be appended in a separate section or in a supplementary report as required.

Quality control must also extend to personnel, and frequent performance evaluations through the use of simulated water samples or proficiency test specimens are recommended (Cada,1974; Gray and Lowe, 1976). It is planned that on a monthly basis in the Ministry of Environment Microbiology Section, well mixed water samples will be divided and distributed among the laboratories as part of the routine workload. By comparing the final results from several technicians, we have become aware in initial testing, of minor discrepancies in incubation temperatures, counting methods and dilution preparation. Proficiency testing should be conducted more frequently during the summer months

when the sample load is heavier and the temporary personnel less trained.

Another aspect of quality control to be considered is laboratory safety. Employees must be made aware of occupational health and safety hazards involved in microbiology. Adequate precautions should always be taken to prevent the spread of microorganisms: such precautions should include swabbing of bench areas with a broad spectrum disinfectant, the use of plugged pipettes and effective decontamination of contaminated media and glassware. A safety checklist should be drawn up with quality inspections and a recording of findings and corrections. An example of a thorough checklist and suggestions for its administration may be found in the paper by Blumberg (1975).

In cases where mobile laboratories play a large part in data gathering, quality control programs should be set up to monitor equipment, media sterility and water supplies. Glassware sterility and media performance could be checked beforehand in the central laboratory.

There is considerable disagreement over the extent of quality control measures necessary. Nagel and Kunz (1973) tested 907 lots of 46 different media and found only 17 to be unsatisfactory. They concluded that "..the employment of quality control methodology may even be detrimental to good practice by unnecessarily diverting limited resources of the laboratory to the testing of media previously certified by a responsible manufacturer." Nagel and Kunz (1973) also mentioned the great cost involved in terms of both money and man hours. On the other hand, Bartlett (1975a) questioned the cost and found that this was offset by the ten to 15 percent reduction in workload brought about by controls on overutilization and production of

clinically irrelevant information. We have found during the past year in the Media-Taxonomy Laboratory that the time and expenditures involved in monitoring the materials and equipment used in the Microbiological Section have saved many hours spent in other years on test repetition and equipment repairs. We can also have considerably more confidence in the results and data reported.

The quality assurance programs applied in the Microbiology Section's Media and Taxonomy Laboratories are organized into four major areas: media, equipment, glassware and water quality. Detailed procedural instructions for each of these programs may be found in the Microbiology Quality Assurance Report including recommendations for the recording and reviewing of quality control data.

November 1976

WATER QUALITY LABORATORY QUALITY CONTROL PROCEDURES

The Ontario Ministry of the Environment laboratories providing routine water quality data take the following precautions to ensure adequate control over the quality of their data.

Primary emphasis is placed on immediate initiation of analysis on the most perishable parameters to minimize effects of sample perishability. Every effort is made to reduce the number of samples outstanding. A weekly report is made on outstanding samples, and weekly graphs are prepared to monitor throughput time.

All results reported are reviewed by comparison against other parameters on the same sample as well as against other samples in the same sample grouping. (Samples are not treated as separate individuals but rather as one of a group received from the same general source, eg. water or waste treatment plant or river survey at the same time.) When considered necessary, previous results from the same source will be consulted to verify unusual data. Repeat analysis is initiated if there is any question about the data.

Since repeat analysis delays throughput time, it is often advisable to initiate analysis on two or more different dilutions to ensure an on-scale result is obtained. This practice is somewhat dependent upon the subjective decision of the particular analyst. When there is doubt, or if the sample is relatively more important, this practice is generally followed.

A particularly important quality control practice exerted when results are being reviewed is the "Ion Balance". When all the major ionic constituents have been analyzed, (i.e. sodium, potassium, calcium, magnesium, (hardness), chloride, sulphate, alkalinity, and in some cases, nitrate) the results are converted from mg/l to milliequivalents/l. The sum of the anions

(negative ions) must then equal the sum of the cations (positive ions) within ± 5%. In practice, the agreement is usually much better than this. Failure to balance suggests error in one or more of the results or instability of the sample usually caused by precipitation of calcium carbonate. This check does not protect against relatively poor precision in the individual constituents, since, on average, precision errors are self compensating. However, when analyzing for these components in drinking water, where precision of analysis is not critical, the ion-balance protects against gross errors.

In the laboratories under discussion here, (Water Quality Section and the Regional laboratories) experience has shown that most of the instrumentation used is relatively stable in calibration and that a certain level of precision is maintained, not because of any special effort, but simply because of the routine nature of the work and the resolution of the instrumentation available. It has also been found that changes in sensitivity can often be traced back to a simultaneous change in the routine; new technician, new standards, new reagents, new or renovated equipment, etc. Therefore, the major emphasis in maintaining data quality has been placed on control of between-run calibration to avoid systematic errors. This has been achieved by application of a two-sample control technique which monitors slope, and when slope is in control, can provide an indication of significant systematic error in the blank or baseline as well. It also provides an indication of the within-run precision to be expected if and when calibration slope is controlled. The system can be used actively on a day-to-day basis to ensure proper calibration of relatively unstable systems; or passively on stable systems just in case something goes wrong.

In order to assess and control accuracy between runs, it has been necessary to formalize procedures for monitoring precision within runs, by requiring a limited number of duplicate analyses each analytical run (generally one out of every twenty samples up to a total of about five duplicates per day). This data is accumulated and used to

monitor and determine within-run precision at various levels of concentration over selected periods of time. It is not presently used in any formal way to control precision.

As a back-up check on precision, as per a recommendation from the IJC, PLUARG program, duplicate samples taken from the source (not a single sample split in two portions) are submitted to the laboratory "blind" on a one in 25 basis. While this procedure includes other potential sources of variability, the information gathered has suggested that in some cases laboratory precision is the limiting factor for routine surface water analysis. This approach has not yet been applied to other Ministry field programs, but does appear to be the best route to take in monitoring the overall effect of sampling and analysis on data quality.

The two-sample A-B technique controls blank as well as slope variability from day to day. Therefore, if a bias is detected via an interlaboratory comparison study it is usually possible to assign a time frame within which this systematic controlled error has occurred. The Water Quality laboratories have participated in all such comparisons initiated by the IJC Data Quality committees as well as those provided by the Canadian federal Quality Control Laboratory at CCIW, Burlington, Ontario.

In order to improve bias control, ampoules containing standard solutions of both single and combined major ion and/or nutrient N and P parameters, prepared by the quality assurance office of the Laboratory Services Branch, are analyzed weekly. Samples spiked with unknown levels of the various parameters are also analyzed on a less frequent basis.

Intercomparisons with smaller laboratories in Ontario have been carried out to determine the compatibility of data. Natural samples submitted over a period of time have been able to provide information on relative precision, blank and slope differences, and have pointed out the effect of sample handling and preservation on data comparability.

Further details on the control procedures are available in the Water Quality Laboratories section of the Data Quality Report Series.

December 1976

QUALITY CONTROL IN THE AIR QUALITY SECTION

A major concern of this laboratory is the possibility of systematic errors resulting from matrix effects. To this end, all non-routine samples and parameters are analyzed by standard additions. If enhancements or depressions greater than 20% are found in the spikes the samples are diluted to reduce the interference and reanalyzed. Analyses with recoveries between 80 and 120% are corrected by the standard additions calculation. In at least 90% of the samples analyzed by standard additions the recoveries are not significantly different from 100%.

As well, during the past year we have completed extensive studies on composite heavy matrix samples, for example, sewage sludge, to determine problem parameters and sample types. Although overall precision and recoveries were excellent, problems were detected and corrected for certain non-routine elements. This work suggests that there are no undetected major systematic errors due to analytical technique.

Our routine quality control procedures, reviewed in January 1976, are restated below. The general Quality Control program applied in a routine fashion in Air Quality consists of:

1) Straight blanks and standards (for equipment calibration).
2) Digested blanks and standards (must agree within ±5% of undigested standards).
3) Replicate analyses (precision data).
4) Round robins (interlaboratory checks).
5) Alternate analysis of same samples by different techniques (inter and intra-laboratory checks).
6) Method of standard additions (spikes, using various compounds and matrices).
7) Standard reference materials.
8) Hi - lo or A-B control samples.

EFFLUENT LABORATORY QUALITY CONTROL - January 1977

Routine Precision:

Each run, which is comprised of up to 40 samples, contains 2 digested blanks, two control samples (A & B) and every tenth sample in duplicate. If the controls deviate from the designed concentrations by more than 20%, the analytical process is stopped, and the source of the error determined. If it is caused by any other than a machine calibration error, the samples are redigested (along with blanks, standards, etc.)

The control data is tabulated and used as a measure of the precision of the analytical procedure.

Routine Accuracy:

Accuracy is estimated by the repeated analysis of standard reference materials and by standard additions studies on "typical" samples. Standard additions studies have been carried out on all routine heavy metals and analytical procedures have developed from this to the extent that there are no significant interferences on routine samples. New or unfamiliar sample types are always analyzed in conjunction with standard additions to minimize or to expose interference or matrix effects.

NBS Bovine Liver and orchard leaves, as well as EPA standard reference materials, are analyzed periodically by our routine methods as a check of our overall procedure. Any significant deviations from the certified values are investigated as to source.

Standards:

Muriel Lawson prepares all the standards for the section. New standards must have a concentration identical to the old. Discrepancies are solved by comparison to standard reference materials and preparation of standards from different starting materials, e.g. Cd metal vs $CdCl_2$.

Instruments:

A log is kept of all operating conditions for each element and each machine. Analyses are not carried out unless the absorbance for a particular standard is within ±10% of the mean of the previously amassed data for that standard.

Participation in round-robins is another means of quality control and is reported separately. Also, testing by a completely independent means of analysis such as anodic stripping voltammetry and/or spectrophotometry greatly enhances the accuracy of most AAS metals data.

MERCURY IN FISH, SEDIMENT AND WATER

The following is a brief statement of quality control procedures currently in use in the mercury laboratory.

Fish Procedure

As with all MOE Hg procedures, inorganic mercuric chloride standards are run prior to beginning digested samples. This generally includes a blank and at least one each of 0.1 ug, 0.2 ug and 0.3 ug standards. The inorganic standard is used until the system is optimized and shows linearity and reproducibility compared to previous day's data.

The digested run includes two blanks, two 0.1 ug, two 0.2 ug and two 0.3 ug methyl mercury standards, and a duplicate aliquot control fish. This is a freeze dried or fresh fish of known mercury value. A spike is added to the first actual fish sample, which is done in quadruplicate. Up until about six weeks ago all samples were analyzed in duplicate with any sample more than ± 10% repeated. This occurred in only twice out of 600 cases; accordingly the use of duplicates was abandoned. All samples are now done singly with every tenth sample in duplicate. Below is a breakdown of the past four months quality control data.

Spiked Samples	High	Low	Mean	%RSD
(Methyl mercury spike)	110%	73%	91.3%	9.6
Wet Control	0.16	0.09	.13	13.8
Dried Control	1.72	1.39	1.57	7.4

A copy of the write-up used for the Hot Block procedure is available. Pages 4, 5, 6, 7, 8, 9 and 10 refer to specific QC procedures used in routine and method development situation.

Water Procedure

Inorganic mercury standards are used as in the fish method. The increased sensitivity of this procedure means that more standards are run before the system is optimized. The digested run includes two blanks, two 10 ng, two 20 ng, and two 30 ng methyl mercury standards. At least once a week a National Bureau of Standards solution is run in duplicate. Samples are done singly. If duplicates or SRM values are off by more than 10% the cause is ascertained and the run is repeated.

Sediment Procedure

Inorganic mercury standards are run as in the fish procedure. The digested run includes two blanks, two 0.1 ug, two 0.2 ug, and two 0.3 ug (methyl mercury), duplicate low control duplicate high control duplicate spikes, and samples are also run in duplicate. This is because of the non-homogeneity of most sediment or soil samples.

For all three systems, fish, sediments and waters, the following are standard procedure:

1) Organic and inorganic working solutions are made fresh weekly from the appropriate stock solution.
2) All equipment is cleaned and maintained (eg. L.D.C. cell is cleaned about twice a week).
3) We participate in as many round-robin or similar comparison studies as possible. An example is provided.

January, 1975 F.R.B. Round Robin

MOE Rexdale	-	#52		#53		#54	#55
		0.22 ± 4%		0.43 ± 5%		0.65 ± 2%	0.84 ± 4%
Average of 22 labs.	-	% RSD		%RSD		% RSD	%RSD
		0.23	14.8% 0.47		12.3%	0.67	10.8% 0.87	9.5%

These data are typical of the results obtained by ITC and most participating laboratories in the FRB and other fish round robins.

4) It should also be noted that on some samples for total mercury, methyl mercury is done as well; this provides a good check for total results, by a completely independent, different technique.
5) Pyrolysis-amalgamation - FAAS is also used in special cases as an independent check on total Hg.

METHYL MERCURY IN FISH

During its routine operation, the quality control on the analysis of methyl mercury in fish could be broken into three general areas:

1) the analysis of standard material and Round-Robins,
2) duplicates, spikes, and standards, and
3) comparison with total mercury results.

1) Standard Material Analysis and Round Robins

Prior to July, 1972, two fish were analyzed with each run (or about 20 fish), and these were termed "High" and "Low" controls. When these samples were exhausted in 1972, several pounds of fish #9794 were homogenized, and analyzed 40 times for standardization. This fish was analyzed as an accuracy check at least once with each run until regular methyl mercury analysis stopped in 1974.

Round robin participation provides an ongoing accuracy check. Methyl mercury analyses have been reported on round robin studies initiated by the EPA, FDA, and five by the FRB. The results have been in the range 70%-100% of the average total mercury results. About 60 such intercomparison fish have been analyzed over the past three years.

2) Standards, Spikes, and Duplicates

Duplicate analyses were usually performed on at least one fish from the previous run as a precision check on a day-to-day basis. Analyses for round robins were also done at least in duplicate.

The methyl mercury method recovers only 85-95% of available methyl mercury. This recovery was determined periodically by spiking studies where several fish, or multiple portions of one fish, were analyzed with and without a methyl mercury spike.

Calibration of the gas chromatograph was done with a solution of methyl mercury in benzene. For a time, aqueous methyl mercury analyses were analyzed with each run, but were abandoned due to the instability of the aqueous methyl mercury standards. Reagent blanks were also included in each run. Calibration was done in duplicate prior to each run, and standards were injected periodically (usually every 5th or 10th injection).

3) Comparison with Total Mercury Results

Every fish sample analyzed for methyl mercury was also analyzed for total mercury, and the results compared. Those samples resulting in methyl mercury results outside 75-125% of the total mercury value were reanalyzed. A statistical summary of almost 600 of these analyses revealed that the methyl mercury averaged 88.9 ± 7% of the total mercury results without correction, with a correlation better than 0.95 between the two sets of results.

The comparison between the total and methyl mercury methods was the most important facet of the quality control program associated with methyl mercury analysis, and provided a check derived from a totally independent method for every result.

NUTRIENTS AND METALS IN SEDIMENTS

Analysis of solid sample types is subject to the usual analytical difficulties associated with liquid samples and in addition are subject to lack of homogeneity or representative sampling and to 'matrix effects'.

Quality control procedures used to detect gross variances due to homogeneity or matrix effects are a) replicate analysis, b) standard additions and c) dilution. These are reviewed below.

Present procedures in the nutrient analyses is to do duplicate analysis of all samples (analyzing for N, P and COD). Included in each 'run' is at least one reference material for which an accumulated average represents the target value. Cell samples where duplicate results exceed a range of ±10% relative to the average of the two values are repeated. This condition occurs less than 10% of the time. Certain samples, which in retrospect would be expected to be less representative or more interference prone, may not agree even after repeat analysis.

It should be unnecessary to continue to carry out duplicate analysis on samples which, from our experience, represent simple matrices such as vegetation. Replicate analysis for every tenth sample should be sufficient in these cases. In fact this condition exists for many sediment samples as a result of the sample preparation procedures (standard sieve sizing, < 2mm and grinding). It is anticipated that, with an initial screening of sample source, many sediment samples could be subject to single analysis with duplicates every tenth.

With regard to metal analysis in these sample types, procedures allow for 'duplicate' analysis every tenth sample in a run (once adequate control has been established), using double the weight in the duplicate sub sample to monitor matrix effects as well as possible sampling effects. Again, use of reference materials within each run is maintained.

It is recognized that the HCl/HNO_3 digestion procedure for metals does not give a measure of true "tota" metal content. The values obtained, however, are thought to be of greater environmental significance. Procedures involving partial dissolution are subject to variability within and between laboratories unless analytical conditions are carefully controlled. This group is participating in inter-lab comparisons examining these factors.

In-House Reference Standards:

Relatively large quantities of samples covering a wide range of composition have been thoroughly blended for use as in-house reference materials. Two portions (0.5 and 1.00 gm) of each of three reference materials are digested in each digestion run of 112 samples for heavy metal analysis. The data is being tabuled for each metal and is used to check for reproducibility from run to run.

Blanks:

At least two and often four reagent blanks are included in each digestion run to check on reagent and glassware contamination. The use of the Mielé washer has significantly reduced blanks. If high blanks are experienced the digestion run is repeated.

Duplicates:

All digestions were in "duplicate" except that 0.5 and 1.00 gm portions were used. Using the "overnight digestion"

procedure, good agreement was obtained both for X on Y and Y on X and it was decided to change to single analysis except for duplicates on every tenth sampled (see R. Bronson: Evaluation of a Rapid Procedure for Heavy Metal Analysis of Sediment and Soil, December, 1975). With change in technician doing digestions and the intention of using spectrographic data for some samples, all samples are again being analyzed in duplicate.

The use of the two weights brought to the same volume of digestate (50 mls) has permitted some indication of the presence of interferences e.g. for calcium and magnesium. These interferences were overcome in this instance by the use of greater dilution using the Auto-Analyzer.

Digestions are repeated if the difference is greater than 15%.

Round-Robins:

The Laboratory has participated in a lead round-robin organized by the MOE. Our in-house reference standards have been sent out for analysis by laboratories associated with the PLUARG program (results not yet available). Satisfactory to excellent results were obtained earlier in several round-robins on dried sludge organized by CCIW. These round-robins covered 12 metals in homogenzied sludge.

NBS Standards:

NBS orchard leaves are analyzed by our perchloric-nitric procedure with very satisfactory agreement for all metals including arsenic. Poor agreement was obtained for NBS fly ash unless pressure digestion with $HF/HNO_3/HCl$ was used.

Spiking Studies:

Spiking studies have been conducted where loss by volatilization was suspected, such as for lead. Recovery of

spiked digested reference samples was 95 and 105% for References 7 and 11, respectively.

Comparison with Results by Different Analytical Methods:

All samples are currently being analyzed both by AAS and Emission Spectrography. Good agreement was obtained for the in-house reference standards and some sediment samples and for some metals. Parallel testing is being continued with a view to using the spectrograph results for at least some metals at higher concentration levels.

Once the ES function is well-defined it is planned to use the AAS and ES systems for corroborative testing.

QUALITY CONTROL OF ANALYTICAL DATA - PESTICIDES

Quality assurance programs for pesticide analysis present special requirements due to the high sensitivity of the instrumentation and the low concentrations investigated. Careful control is required to guard against contamination of the samples.

Sampling

Valid sampling procedures are essential, but will not be discussed here, except to mention that special solvent-rinsed containers must be used and that these must be fitted with caps lined with Teflon.

Background levels

Reagent blanks must be run with every set of samples to ensure that no interferring contaminants are introduced into the samples. Glassware should be verified regularly at the end of the washing-drying cycle, before being used. Every new batch of solvents or reagents must be checked for impurities prior to use.

The reagent blanks should show no background levels, when analyzed under conditions corresponding to the "detection limits" of the procedure.

Recoveries - Spikes - Duplicates - Controls

Recovery studies are meaningless at the extraction stage since spiking a substrate cannot compare with a "real" sample where the pesticides have been adsorbed into the matrix over a period of time. Such studies are valid, however, for the clean-up and determination stages and should be conducted whenever a modification is brought to bear on any part of the procedure.

So far as water samples are concerned, the whole sample is extracted, so no duplicate aliquots can be taken. Each daily set of samples (16) must include a reagent blank and a "spike", using a common batch of reagents and solvents. The spiking standard should be within the normal working range and should be added to tap water.

Duplicate aliquots of a fish sample are difficult to evaluate quantitatively unless the original sample contained tissue only and was homogenized thoroughly and mixed perfectly. The same applies to soils and sediments which must be thoroughly mixed before duplicate aliquots can be taken.

The accuracy of analytical procedures for fish and sediments can be verified by use of a "control" sample prepared by thoroughly mixing several "real" samples. An aliquot of this "control" should be run along with a reagent blank and one duplicate sample with every daily set of samples (18), using common reagents and solvents.

Gas Chromatography

Gas chromatographs must be under continuous surveillance to ensure maintenance of optimum operating conditions. At the start of each day, standard solutions must be injected at various concentrations to check the performance of the instrument and to calibrate it. Standards should then be injected at regular intervals (2 hours or 4-6 samples) to verify reproducibility.

At trace levels, problems are encountered in the positive identification of residues. Identity of the residue is obtained by comparison of retention times, so that confirmation should be done by using another column yielding a drastically different relative retention time (Raldrin etc.) for the residue in question. A different detector which would respond to a specific element (i.e. N, P. or S detector) in the compound could provide good confirmation. Chemical conversion yielding a specific

derivative is a good confirmatory procedure and should be used whenever unexpected residues are found.

The most viable technique for confirmation is G-C-Mass Spectrometry when sufficient amounts of compound are available (several samples can be combined).

January 1977

ORGANIC TRACE CONTAMINANT SECTION

Q U A L I T Y A S S U R A N C E

Quality assurance for the OTC Section present unique problems, due to the wide range of concentrations of contaminants that may be present in samples, and the large number of different organics that may be present, as more than 2 million organic chemicals are known to exist.

Sampling

Although sampling is rarely done by OTC staff, the sampling techniques, preservation, transport and sample handling are discussed constantly with the samplers.

Blanks

Reagent blanks are run when analyzing for trace organics to ensure that there are no contaminants from the laboratory introduced into the samples.

Sample Preparation

Organic contaminants are extracted from a number of matrices. Experiments are run to determine the most efficient method of extraction of specific organics from a given matrix.

Replicates

With water samples, the whole sample is extracted, so duplicated samples must be made available from the samplers.

With air particulate analysis, in many instances, only a portion of the filter is made available. Sample homogenicity is assumed, but substantial errors may be introduced if the filter is nonhomogeneous. Duplicates are made available when possible.

Standards

Standards are routinely checked for authenticity by nuclear magnetic resonance, infrared, ultraviolet or mass spectrometric analysis prior to routine use. The standard purity is verified by gas or liquid chromatography.

Instrumentation

Identifications of trace organics are done primarily by comparison retention times on gas or liquid chromatography. Standards are to run at frequent regular intervals to ensure reproducibility.

Retention times of the organics under investigation are routinely checked on two different chromatographic columns to confirm their identities. Different detectors which respond to specific groups (e.g. halides, chlorides, sulphur, phosphorous) in an organic compound also provide confirmation of their identities.

Before a specific analysis goes "on-line" the chromatographic peaks in question are verified by gas chromatographic-mass spectrometric analysis.

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QUALITY CONTROL PROCEDURES FOR IDENTIFICATION OF ORGANICS BY MASS SPECTROMETRY

The Organic Trace Contaminants Section receives samples requiring screening for (specified) potential organic contaminants. The nature of the samples received usually requires the development of special extraction and recovery techniques suitable to the particular sample. Therefore, the analyst plays an important role in assuring data quality.

Since the usual need is for identification rather than quantification the concepts of precision and accuracy are not as relevant as those of specificity and recovery. In order to confirm negative results, for instances, the sample can be spiked with the suspected constituent to ensure that the sample treatment processes will permit its recovery and identification. The control steps taken to maintain the mass spectrometers in correct working order are described below. If quantification is needed, gas-liquid chromatographic techniques would be employed as for pesticide residue analysis.

1) Dupont 491BR Mass Spectrometer (GC/MS system)

Daily; Mass range of instrument is tested with a high molecular weight (>750) fluorocarbon, perfluorokerosene. The instrument is then calibrated by matching the mass indicator with peaks from the perfluorokerosens which appear over the mass range of m/e 69 to m/e 331.

The sensitivity of the instrument is also monitored daily by noting the height of the peak at m/e 69 vs amount of perfluorokerosene injected and gain factor.

Monthly; Absolute sensitivity of the GC/MS in coulombs/microgram is tested by recording the molecular ion of methyl stearate (m/e 298) as a standard solution of the chemical is injected on the gas chromatograph.

QUALITY CONTROL NEEDS IN ASBESTOS ANALYSIS

Asbestiform mineral fibres have been widely used in the manufacture of industrial and domestic products since the turn of the century. Public concern over the environmental and health effects of these fibres has led to a need for reliable, quantitative, sampling and analytical procedures. Although several different instrumental techniques have been developed, the analysis of environmental samples by transmission electron microscopy is currently the preferred method. However, inter-laboratory comparison studies conducted by several government agencies have demonstrated significant differences between laboratories analyzing 'identical' samples of air or water. There are several reasons for this, generally related to difficulties in obtaining representative samples, in recovering fibres from the sample taken, in sample preparation and dispersion for counting, and in identification and analysis procedures.

Therefore, a comprehensive quality assurance program would have to examine several areas of concern in order to determine their importance and to develop suitable control procedures. Some of these would be;

a) sampling equipment and technique.
b) filtration efficiency for different types of air filtration systems and filters.
c) container types, lid liners, etc., for water sampling.
d) uniformity of fibre distribution on filters or in water samples.
e) stability of fibres during storage.
f) effect of filtration of air and water on fibre loss or gain by splitting of fibre bundles.
g) effect of drying, low temperature ashing or filter dissolution using organic solvents, and other preparative techniques, on fibre recovery.
h) uniformity of fibre deposition on electron microscope grids.
i) fibre identification and enumeration procedures.

2) AEI MS-30 Mass Spectrometer (ADP/GC/MS system)

Daily; Mass range of the instrument is calibrated after injection of perfluorokerosene reference compound. Computer matches the observed fragmentation pattern with a stored reference fragmentation pattern. Internal calibration is then adequate for 24 hours or more of operation with every mass correctly identified automatically by the computer. The sensitivity of the instrument is monitored by noting the height of the fragment peak at m/e 181 vs amount of perfluorokerosene and gain factor.

Monthly; Absolute sensitivity of the GC/MS system in coulombs/microgram is tested by recording the molecular ion of methyl stearate as a standard solution of the chemical is injected on the gas chromatograph. The absolute sensitivity of the MS alone is tested by the direct evaporation of cholesterol in the source whilst measuring the molecular ion at m/e 386.

January, 1977

A committee of asbestos analysts, organized by the Ontario Ministry of the Environment, is presently examining these and other variables in order to develop a reliable, quality controlled procedure for analysis of asbestos in air and water samples.

Further information can be found in an interim report describing the "Determination of Asbestiform Mineral Fibre Levels in Air and Water".

January 1977

QUALITY CONTROL PROCEDURES FOR EMISSION SPECTROSCOPY

In emission spectroscopy (ES), the multiplicity of analytical channels, excitation modes, and sample matrix types requires that a great deal of attention be given to quality control. This is both assisted and complicated by the computerized direct readout system. Areas of primary concern would include:

1) Internal standardization to compensate for variation in excitation energy.
2) Spectroscopic buffers to reduce sample matrix effects.
3) Background channels to monitor the variation in background emission and detection response with wavelength.
4) Calibration curve analysis to determine the gain and offset factors to be expected for certain matrices.
5) Standards to confirm the stored calibration curve.
6) Blanks to correct for reagent and sample preparation contamination.
7) Analysis of reference materials to confirm recovery.
8) Line profiling to determine proper detector alignment with the desired spectral line and to detect defective photo-tubes, or amplifier circuit boards. Control of room temperature and humidity provides more stable operating conditions to prevent spectral line and/or computer electronics drift.
9) Matrix effect evaluation to determine the presence and/or effect of interferences via standards, spikes and comparison against results obtained by alternative procedures (eg. AAS).

10) Parallel evaluation of ES and AAS techniques based on certain data quality objectives in order to determine which technique is more suitable for specific parameters, sample matrices or data needs.

11) Duplicate analysis using two different weights of digested sample provides precision data as well as providing a reading if the calibrated range is exceeded by the larger sample.

12) Photographic plate recording of contradictory results or problems are encountered in the direct reading mode.

13) Diagnostic programs for the computer which can be used to determine the stability of individual channels in the direct reading mode. Ten analytical runs are made with the source either on or off. Exceptionally high standard deviations point to improper function.

A more detailed description of the procedures used is in preparation. Queries may be directed to Dr. M. M. Moselhy, Emission Spectroscopy Laboratory.

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